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AN INVESTIGATION OF IMPROVED SAMPLE HANDLING PROCEDURES IN ULTRASONIC NEBULIZATION TECHNIQUES

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Tomorrow Enterprises

TECHNICAL REPORT AFML-TR-68-354

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AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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FOREWORD

This report was prepared by Tomorrow Enterprises, Portsmouth, Ohio on a sub-contract with the University of Cincinnati under USAF Contract No. F33615-67-C-1565. The contract was initiated under Project No. 7360, "Chemical, Thermal, and Dynamic Properties of Materials", Task No. 736005, "Compositional, Atomic and Molecular Analysis". The work was administered under the direction of the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio with Mr. Freeman F. Bentley and Mr. James H. Muntz as Administrative and Technical Project Engineer, respectively.

This report covers work performed from April to September 1968.

The manuscript was released by the author in October 1968 for publication.

This technical report has been reviewed and is approved.

Freeman F. Bentley

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ABSTRACT

An ultrasonic nebulizer based on flowing a sample solution across the vibrating face of a piezoelectric crystal produces an aerosol useful for instrumental analytical techniques. The piezoelectric element is coated with a ceramic glaze resistant to many analytical solutions. Different and better coatings will be needed, however, for especially corrosive solutions. Ultrasonic nebulizers inherently more resistant were designed but had limited operational life and were not perfected during this study.

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I. INTRODUCTION

The value of ultrasonic nebulization for the introduction of liquid samples into a plasma arc for alloy analysis was established in the project reported as AFML-TR-67-400. The apparatus developed during that project utilized a "batch process" precluding its application to continuous or "on-stream" analyses. This investigation attempted to utilize ultrasonic "flash nebulization" in which solutions are not recycled and continuous operation can be attained without inter-sample contamination.

II. SUMMARY

A new ultrasonic nebulizing device, developed during this investigation, produces useful aerosols from sample solutions continuously presented to its vibrating face. The transducing element is mechanically loaded and electrically driven from its back side only. The working face of the vibrating element is protected from the mple solution by a coating of low-melting-point glass. The corrosion resistance of the glass now limits the analytical usefulness of the device. The new design, however, is applicable with coatings of increased chemical resistance.

Only short-lived devices resulted from attempts to fabricate displaced-impingement-point, direct-impingement nebulizers with the corrosion resistance of the nebulizer detailed in Report AFML-TR-67-400. Operable designs were limited by material failure under the high power conditions required.

III. DIRECT-IMPINGEMENT ULTRASONIC NEBULIZER

A stated aim of this project was to design a direct nebulization assembly, based on the design of Figure 1, exploiting ceramic-coated piezoelectric transducers. While the specific chemical resistivity of the coatings were not known to us, we felt that their use represented the best theoretical approach to the overall sampling problem. Our immediate goal was to design a mechanical assembly which would permit these coated transducers to operate effectively, efficiently, and lastingly. Hopefully, concomitant encouragement of commercial development

of more resistant glazes and study of coating problems would permit eventual upgrading of analytical utility.

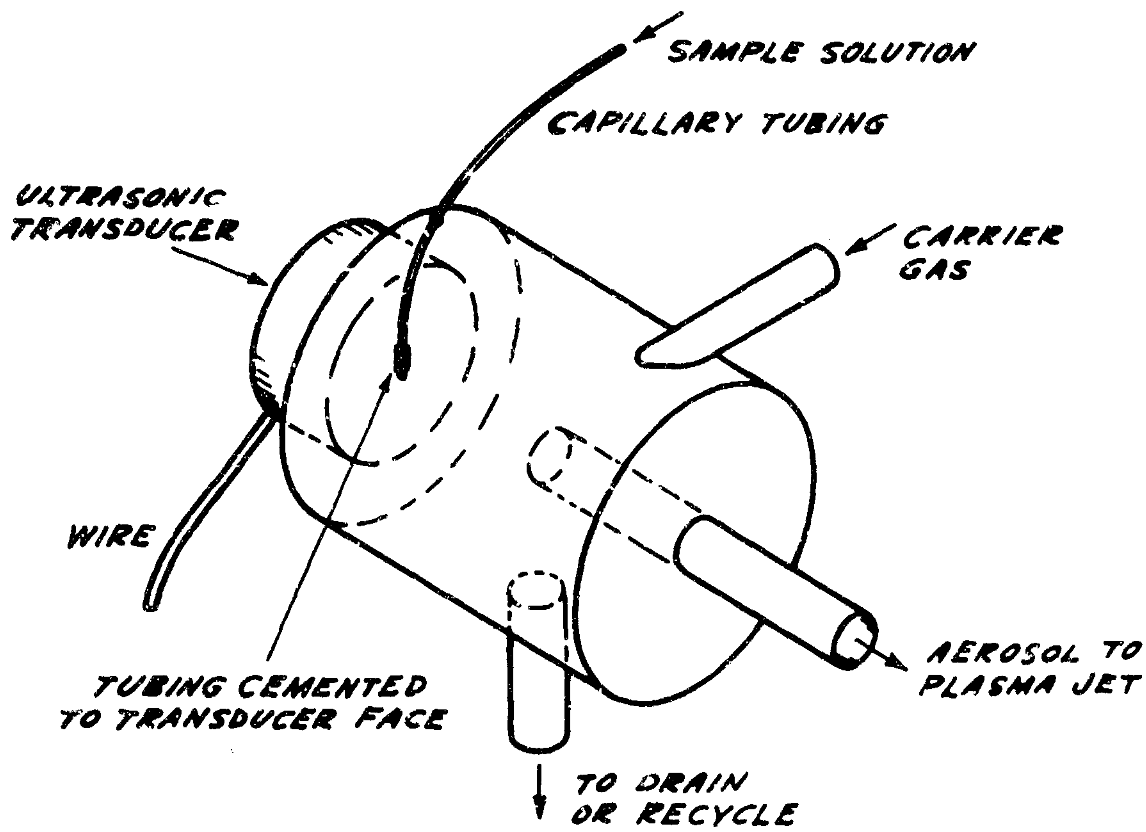

FIGURE 1 ULTRASONIC NEBULIZER-DIRECT IMPINGEMENT

A. Development

We obtained a number of glazed, high frequency, transducer elements from commercial sources. Characteristically, for processes more art than science, it was difficult to obtain meaningful specifications from their manufacturers. Also characteristically, the coatings proved considerably less chemically resistant than implied by their manufacturers. Of the elements purchased, our principal efforts were with Gulton Industries' HD-11 discs, 0.875" diameter, 1.4 megahertz, coaxial electrodes and blue glaze coating, Code 1136. About half the batch purchased were imperfectly coated and could not be used.

Effective, long-lasting mounts for the transducing discs were difficult to fabricate. Effectiveness is a function of mechanical loading and electrical

impedance matching, also somewhat dependent on mechanical parameters. Longevity is directly related to effectual cooling which must be accomplished without loss of electrical power. The final design for this project, Figure 2, is traceable to fruitful discussions held with Dr. Martin Greenspan, head of the Acoustics Section, National Bureau of Standards.

B. Construction Details

The mounting of the piezoelectric transducer disc is illustrated in the detail sketch of Figure 2. The working-face electrode extends beneath the glaze, over the edges of the disc, and appears as an annular ring electrode around the outer edge of the back surface. It is electrically contacted by a copper tube, 22 mm in diameter with a 2 mm wall, which is cemented to it with electrically conductive epoxy adhesive. The opposing electrode is a silvered spot, 13.5 mm in diameter, which is centered on the back surface of the disc. This spot is contacted by a water-cooled brass subassembly also held by electrically conductive epoxy. The volume between the copper tube and brass piece is filled with thermally conductive, electrically insulating epoxy to permit overall cooling without power loss. Secondary electrical connections are made with a coaxial connector, JAN UG-1051/U.

The semi-mounted transducer is further mounted by casting into a plug of methyl methacrylate which slides into the chamber used to capture and direct the aerosol produced in operation. The sample solution is fed to the transducer by a TEFLON tube, 1/16" od and 1/32" id. The tube rests lightly against the working face of the transducer.

IV. DISPLACED IMPINGEMENT POINT - DIRECT IMPINGEMENT NEBULIZATION

During this project period we also investigated extensively improved designs of a nebulizer configuration previously proposed, Figure 3. The work was motivated by the possibility of constructing units with materials resistant to solutions containing fluoride ions. Glass-like coatings on the glazed disc devices were obviously unsuited for such service.

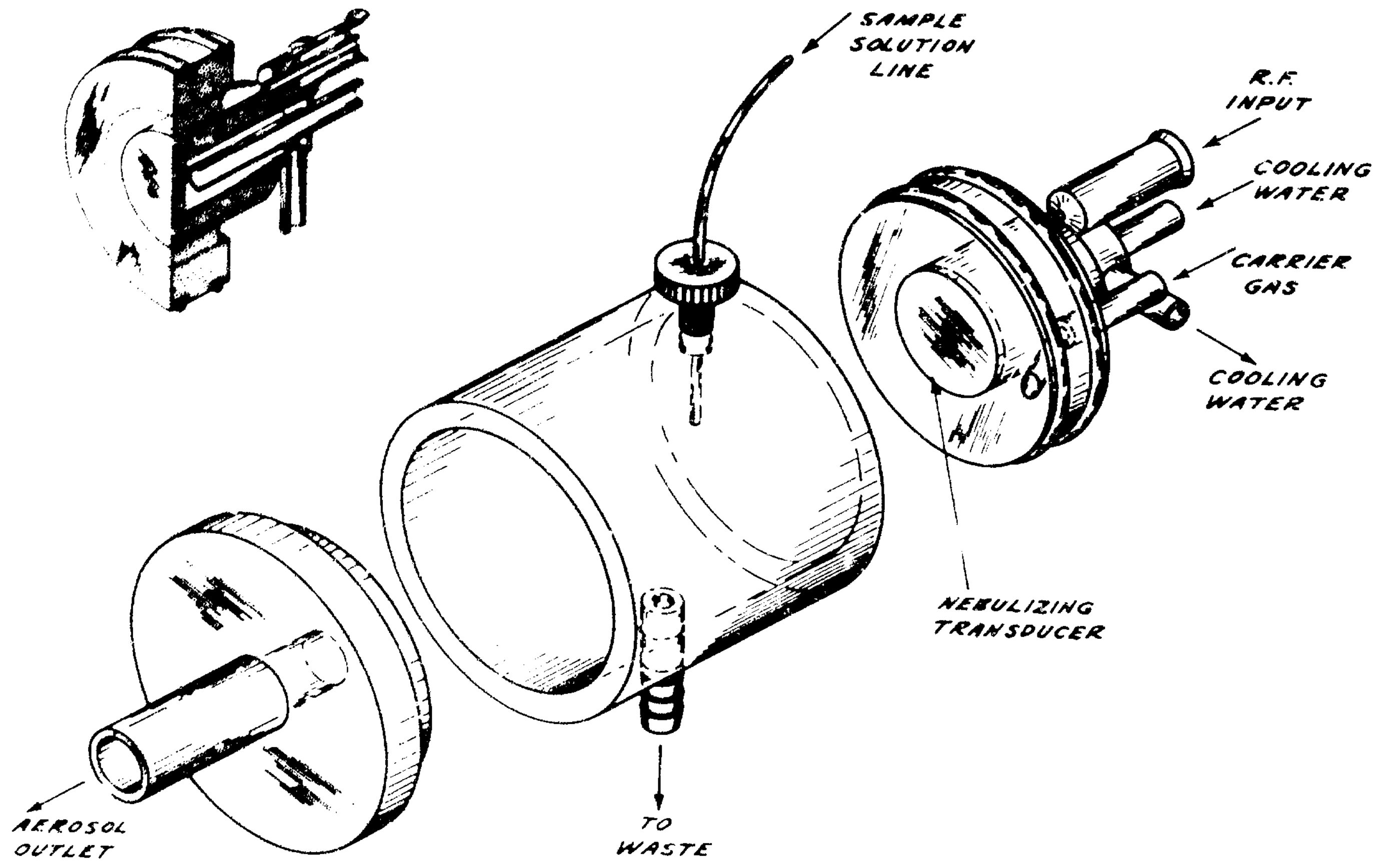

FIGURE 2 NEWLY DEVELOPED DIRECT IMPINGEMENT ULTRASONIC NEBULIZER

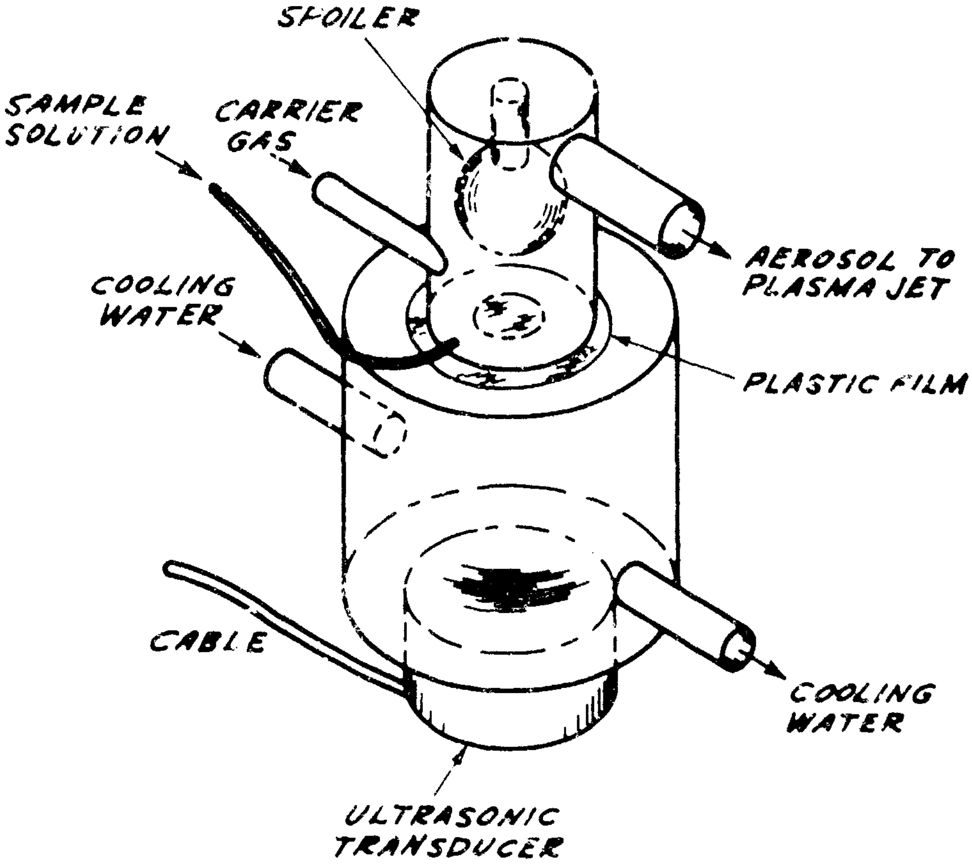

FIGURE 3 ULTRASONIC NEBULIZER-DISPLACED IMPINGEMENT POINT

For the new studies we obtained custom, cupped-disc transducers, 2" diameter, 3" radius of curvature, 3 megahertz air resonance. These discs could be driven very hard in our water cooling-coupling chamber without damaging the crystal. Several of these discs were fabricated into complete assemblies having small sample volumes for continuous operation under non-recycling conditions. Essentially all of the assemblies functioned well - for a short time, five to twenty minutes. The high power densities required for adequate insonation perforated the interfacial plastic films. Then too, in spite of their excellent optical figure, the focusing transducers, reacting to non-fundamental frequencies in their drive power and to non-Z axis couplings produced acoustical waves not focusing to a point. When the sample volume was made as small as desired, the sample-confining walls absorbed enough acoustical energy to become deformed.

The resulting materials problem is a temporary, not absolute, obstacle to the utilization of this particular configuration. There is no theoretical reason why it can not be a useful design. Another important requirement, aside from materials selection, is the use of rf power supplies with cleaner output waveforms.

V. ALLIED INVESTIGATIONS

A. Various Piezoelectric Compositions

A test mounting for the comparison of various piezoelectric compounds was assembled as part of the reported studies. Four different compositions were custom pressed into flat discs for our evaluation. As anticipated from basic engineering data, we found three materials better suited to project use than the older compounds more readily available from commercial producers. We have requested additional test discs of superior composition. It is apparent that this work is encouraging manufacturers to promote the use of the newer formulations.

B. Operation at Various Frequencies

The test assembly just mentioned was also designed to permit comparisons of piezoelectric discs sized for different frequencies at resonance. Elements, otherwise similar, were purchased for the frequency range 0.8 to 5 megahertz. Complete tests during the project period were stymied when it became obvious that at least three separate power supplies must be used for valid comparisons. The supply impedances must be closely matched for optimum drive and requirements are markedly different over the frequency range of interest.

C. Pumps

Continuous nebulization of analytical solutions inherently requires low flow rate pumps with considerable chemical resistivity. Nonpulsating flow is also desirable. Our search continued during the study period for commercially available pumps meeting our requirements. As part of this search, we were able to borrow or test in a distributors' facility highly touted pumps advertised to have special features. Our search goes on.

The continuing introduction of new pump designs is indicative of an extensive need in the scientific community for pumps actually delivering the advertised performance of present units. Tomorrow Enterprises is, fortunately although somewhat reluctantly, accumulating design knowledge which will conceivably result eventually in an adequate pumping system.

VI. CONCLUSIONS

A direct impingement ultrasonic nebulization device for the introduction of liquid samples into a plasma jet was designed for use with coated transducers. While presently available coatings are not resistant to all solutions of analytical interest, the design of the new device can be used with improved coatings as they are developed. It is the first design with such capability.

VII. RECOMMENDATIONS FOR FUTURE WORK

Improved versions of the coated transducer type of direct impingement ultrasonic nebulizer can undoubtedly evolve from continuing operational experience with the present device.

Development of the displaced-impingement-point, direct-impingement nebulizer is dependent upon careful selection of construction materials and the development of better rf power generators.

Transducer elements with more resistant coatings need development while compositions of greater strain response must be utilized even with present coatings.

The analytical significance of higher frequency sample insonation should be established. Concurrently the most efficacious exploitation of aerosol samples in plasma jet excitation should be determined. This development may well require modification of existing jet designs to make high efficiency excitation zones available for spectroscopic viewing.

Development effort on adequate solution-pumping subsystems is a requirement for obtaining full value from any nebulizing device in a complete sampling system for instrumental analytical techniques.

VIII. TECHNICAL PRESENTATIONS

Information from these studies was used, in part, during a lecture-demonstration entitled "The Ultrasonic Nebulization of Solutions for Instrumental Analytical Procedures" given October 21, 1968, during the 15th Spectroscopy Symposium of Canada held at Toronto, Ontario.

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